Tapered Optical Fiber Coated with ZnO Nanorods for Detection of Ethanol Concentration in Water

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ABSTRACT— This work presents ZnO nanorods coated multimode optical fiber sensing behavior in response to ethanol solution. The sensor operates based on modulation of light intensity which arises from manipulation of light interaction with the ambient environment in sensing region. For this purpose, two steps are experimentally applied here; etching and then coating fiber with ZnO nanorods to provide stronger evanescent waves causing an enhanced interaction. Long length of fiber (15 mm) was etched uniformly and then well-ordered ZnO nanorods were grown hydrothermally on the core of optical fiber. Fiber coated with ZnO demonstrated an enhanced sensing performances such that response time decreased to 0.6s, linearity increased to 97% and sensitivity improved. Applicable features of the proposed device such as fast response time and high linearity make it favorable candidate for fiber optic sensing applications.

KEYWORDS: Evanescent field, Ethanol sensor, Hydrothermal, Optical fiber sensor, ZnO nanorods.

I. INTRODUCTION

Nowadays real-time and in-situ detection of ethanol with high sensitivity is of great importance in many industrial and biological applications. Among different sensing approaches, the efficient technique in determination of alcohols is based on optical devices. Optical sensors possess inherent advantages of safety, miniaturization and cost-effective for demanding analytical applications. A number of ethanol sensors have reported based on Fabry-Perot cavity [1], fluorescence [2-6], Raman spectroscopy [7] and optical fibers [5, 8-12]. Owing to their intrinsic advantages, fiber optic sensors are an ideal candidate and have been employed for ethanol sensing [8, 13, and 14]. In this regard, Penza et al. [10] deposited single-walled carbon nanotube on optical fiber through Langmuir-Blodgett technique for ethanol sensing application at room temperature. In another study, Zhengtian et al. [12] investigated response of long period fiber coated with SnO2 thin film to ethanol. Fiber optic sensor overlaid with metal oxide nanostructures (TiO2, ZnO and SnO2) exhibited enhancement in sensor performances due to high surface to volume ratio and applicable chemical and physical properties [11, 15-18]. More specifically, ZnO nanostructures with a direct wide band gap around of 3.2 eV at room temperature, reactive surfaces for chemisorption [19, 20] and large exciton binding energy (~ 60 meV) render it as a suitable choice for sensor fabrication. Among various morphologies of ZnO, one-dimensional structure is of exclusive feature of high surface-to-volume ratio and is relatively simple to grow in aqueous solutions due to the polar nature of the (002) crystal plane [21]. Different methods have been devoted to synthesize ZnO nanorods, including physical and chemical vapor deposition [22, 23] and vapor transport [24]. These methods require a relatively high temperature and complex vacuum environment during the growth process. On the other hand, hydrothermal method of growing ZnO nanorods is simple, environmentally friendly and energy-efficient [25, 26]. In recent decades, hydrothermally growing nanorods on the flat substrates have been studied intensively [27-30]. However, few published works in the literature investigated the well-arrayed dense nanorods on the curved surfaces such as optical fiber [15, 31]. In evanescent wave based fiber optic sensors a certain ratio of the transmitted light through the fiber is leaked out by removing a certain region of the cladding of the fiber which is known as evanescent waves. In the modified cladding region, the evanescent wave trapped through the coated nanostructure provides the interaction with the external medium. This technique was exploited for developing evanescent wave absorption based sensors. Absorption of the evanescent field occurs when the nanostructure medium, which forms and modifies the cladding of the waveguide, enable to interact with the analyte [32-35]. Herein variation of refractive index corresponding to different concentration of analyte will affect the absorption of evanescent wave in the sensing region. Since ZnO coated optical fiber is able to interact with the ethanol solution giving rise to the manipulation of output intensity and providing a highly sensitive sensor.

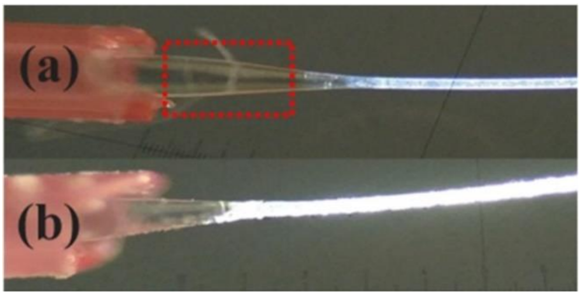


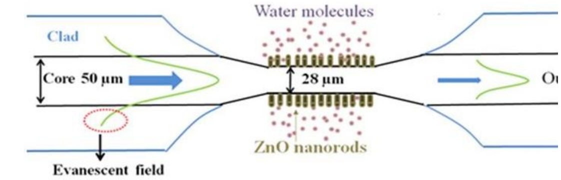
Fig. 1. Microscope image of (a) bare optical fiber etched to 28 µm in diameter, (b) ZnO coated fiber with same diameter.

Presence of nanostructures with higher refractive index than optical fiber provides more evanescent waves on the sensing region that improve and facilitate light interaction with ambient medium. In the present paper, large-scale arrays of highly ordered ZnO nanorods were grown hydrothermally on the etched multimode silica fiber. The behavior of thinned bare fiber was compared with ZnO coated fiber in terms of evanescent wave generation and sensing performances. Modes distribution in fiber core under different refractive index around ZnO coated fiber was also studied theoretically.

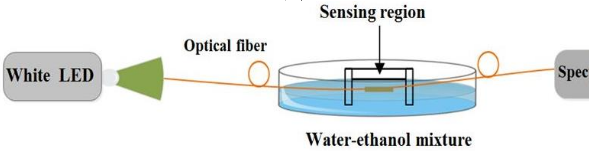
II. EXPERIMENTS

A. *Sensing Principle and Sensor Setup*

The proposed sensor operation is fundamentally tied to the variation of the fiber surrounding environment refractive index. By changing the concentration of gas or liquid, the refractive index around sensing region would change which leads to variation in output light intensity. Since these fashions of sensors work in the principle of evanescent waves, the key point for designing high sensitive sensor with fast response is providing high evanescent field in sensing region [36]. Thinner fiber resulted in a stronger interaction for sensing applications; more evanescent wave can be produced by higher light leakage in tiny fibers. In this regard different methods have been reported including tapering fiber through flame heated method [15], mechanically removing clad of fiber [37, 38] and chemical etching [39]. In this work, long length of fiber thinned with high smoothness through low cost chemical etching method. Referring to our recent experiment [36] the efficient evanescent waves attained for multimode fiber with 28 µm in diameter, near 76% of coupled light leaked out the fiber core. It should be noted that utilizing nanostructure with higher refractive index than fiber core allows higher light leakage. As shown in Fig. 1 the effect of etching fiber and growth of nanorods in generation of evanescent field is demonstrated experimentally. Figures 1(a) and 1(b) illustrate evanescent field, the emerged light around the fiber, for the bare and the ZnO coated fiber, respectively. It is clear that in the beginning of cone shaped region (as shown in dotted rectangular in Fig. 1(a) light didn't leak out, while in the thinner region it is able to leaked out as evanescent fields. ZnO coated fiber allowing higher portion of light leaked out of the fiber core and propagated in modified clad (ZnO) region.



(b)



(b)

Fig. 2. (a) Sensing region (b) Experimental setup in ambient conditions for real-time monitoring of transmitted light intensity through optical fiber during alcohol sensing.

This phenomenon provides more interaction of light with external stimulus. ZnO (n~ 1.9) with higher refractive index than silica core (n ~1.45) operates such as waveguide, moreover it can chemisorb and physisorb different kind of analyte applied as a suitable candidate for sensing applications.

Figure 2 shows the experimental setup of optical fiber sensor that immersed in water-ethanol mixture with different concentration. It consists of LED light source (model: HNK-HP003-E45) with wavelength emission ranging from 400-800 nm coupled to the multimode fiber. It should be noted that according to our previous experiment [40], maximum intensity variation occurs at wavelength of 560 nm so the rest of sensor measurements and calculations were done at this particular wavelength. The light emerging from the other end of the fiber is fed to a spectrometer (model: HR 4000 CG-UV-NIR). The signal from spectrometer was transferred to computer and output intensity corresponds to each concentration was recorded. It should be noted that real-time monitoring of transmitted light intensity inside optical fiber was done through high resolution spectrophotometer to precisely obtaining sensor time responses. Once sensing region immersed in solution, air around the fiber replaced with water-alcohol molecules, two different responses observed for bare and ZnO coated fiber. In the former, noticeable increase in output intensity was observed. Hydrophilic property of silica fiber core leads to back reflection of leaked light into the core causing increase in output intensity [41]. By increasing alcohol concentration and so increasing surrounding refractive index than that of pure water, output was decreased in consistence with theory and higher portion of light leaked out. For the latter case, water molecules chemisorbed on the ZnO nanorods surface and there was no more contact between fiber and water molecules so by increasing alcohol concentration output light intensity decreased.

B. *Sensing Region Preparation*

Sensing region prepared through chemically removing the cladding of multimode fiber (core/cladding diameters of 50/125 µm and numerical aperture of 0.2 ± 0.015) up to the core. Middle segment of fiber, with 15 mm length, immersed in the diluted 25% HF (HF 40%, E. Merck) acid solution, such that the average etching rate for cladding removal was about 0.75 µm/min and it was about 1.16 µm/min to decrease fiber core diameter. Fiber waist diameter etched to 28 µm and then rinsed with deionized water. ZnO seed particles were uniformly covered silica fiber as a substrate by dipping the fiber into the seeding solution [36] for 10 min and then annealed at 150 °C in the oven for 20 min to evaporate the solvent, this process repeated three times. ZnO nanorods obtained after hydrothermal treatment in autoclave at 95°C for 5 hours. Equimolar aqueous growth solution (0.01M) of zinc nitrate hexahydrate (Zn (NO3).6H2O, 99% purity, E. Merck) and hexamine (HMTA, 99% purity, E. Merck) (C6H12N4) dissolved in 100 ml deionized water and poured in Teflon-lined sealed autoclave. Finally, the fibers were taken out of the growth solution and rinsed through deionized water carefully followed by baking at 150 °C for 1 hour.

III. Results and Discussion

A. *SEM Analysis*

Figure 3 (a, b) illustrate SEM images of the bare optical fiber and seeded fiber covered with ZnO nanorods. Bare fiber etched uniformly with long-length, providing applicable substrate for vertical growth of nanorods and sensing applications. Over laid fiber exhibit well-arrayed and relatively high density of ZnO nanorods pointed outwards from the fiber surface with aspect ratio more than 8 and average length/diameter of 2 µm/220 nm. Fibers seeding with ZnO nanoparticles establish nucleation sites and lowering the thermodynamic barrier resulting improvement in aspect ratio and density [42]. Employing equimolar solution of zinc nitrate and hexamine in hydrothermal treatment causes growth of ZnO rods with hexagonal wurtzite structure [42].

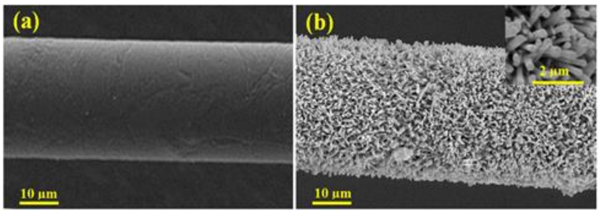


Fig. 3. SEM images of (a) etched optical fiber and (b) Seeded fiber covered with ZnO nanorods (inset shows enlarge view).

B. *PL Analysis*

PL analysis is a powerful study that exhibit the absorption of energy and subsequent emission of light under the term luminescence providing noticeable knowledge related to the material defects originated during growth process. Figure 4 shows the room temperature photoluminescence (PL) spectra obtained from the silica optical fiber covered by ZnO nanorods. Since coated optical fiber was too thin, to better observe the emitted luminescence several identical coated fibers put in the sample holder and excited at wavelengths of 240 nm. It can be seen that samples show a typical luminescence behavior with the two emissions of a strong UV peak around 397 nm and a broad deep level peak with green emission in the 490~560 nm region. The UV emission is due to recombination of free-excitons through exciton-exciton collision process [43]. It has been suggested that green band emission is related to singly ionized oxygen vacancy in ZnO [44, 45], resulting from recombination of a photo-generated hole with the singly ionized charge [46]. Oxygen vacancies and zinc interstitials, point defects, are critical characteristic in sensing applications as they cause very large variation in the surface conductivity [47]. Changes in charge density have direct impact on optical properties, namely refractive index of nanorods altered correspondingly [48].

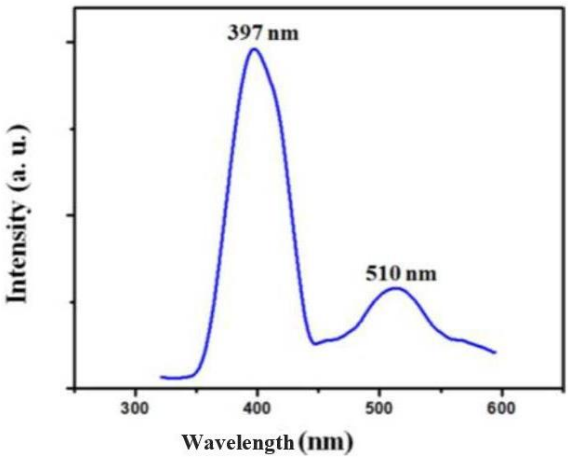


Fig. 4. Room-temperature PL spectrum of ZnO nanorods that are grown on the optical fiber excited at wavelength of 240 nm.

C. *Sensor Response to Ethanol*

To investigate sensor response to different ethanol concentration, sensing region was immersed in ethanol-water mixture for about 2 min and was withdrawn out and stayed in air for a few seconds this process repeated consecutively for different concentrations. Range of ethanol concentration in distilled water was from 0-80 volume percent. First, fibers immersed in pure water, as mentioned above bare fiber exhibited different behavior compare to ZnO coated fiber. The output intensity plotted as a function of ethanol volume percentage (Fig.5 (a, and b)). Transmitted light intensity reach to its stable value after 0.8 s for bare fiber and 0.6 s for ZnO coated fiber as immersed in solution.

When the fiber was withdrawn out from solution, spiky signal appeared and proceed towards the initial value (that of in air). Since residual of solution's droplets exist on the fiber surface, the transmitted light couldn't return to its initial value. Herein, we tried to exhibit sensors ability to consecutive measurements and its distinction accuracy for solution with different concentration.

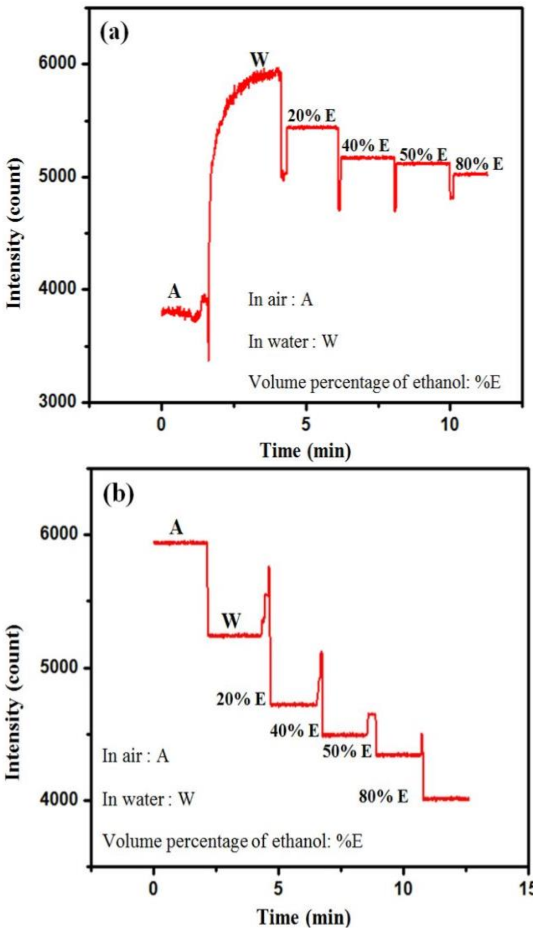


Fig. 5. Change in output intensity upon exposure to different ethanol concentration versus exposure time for (a) bare fiber and (b) ZnO coated fiber.

As mentioned above, bare and ZnO coated fibers immersed in ethanol solution with different concentration. In order to investigate the sensitivity and linearity of the sensor response, the output light intensity against ethanol concentration of was considered as illustrated in Fig. 6. The output intensity decreased by increasing ethanol concentration. It can be seen that sensitivity (defined as slope of fitted line) enhanced from -10.69 (count/%) for bare fiber to -14.94 (count/%) for ZnO coated fiber. Sensor linearity improved from 87% for bare fiber to 97% for ZnO coated fiber. Minus sign in sensitivity refers to decreasing in transmitted light intensity by increasing in ethanol concentration. These intensity variations in tapered optical fiber are described by the lambert-Beer Law [17, 49] as shown by the following equation:

$$I = I_0 e^{-\gamma l}, \gamma = \alpha_m r_f C \qquad (1)$$

where I and I_0 are the light intensities before (I_0) and after (I) interaction of sensing region with analyte. The values C and l are the concentration of analyte and length of tapered fiber, respectively. The parameter γ stands for evanescent wave absorption coefficient, which is the function of concentration, effective fraction of the total guided power (r_f) in the sensing region and α_m that is the bulk absorption coefficient of the absorbing material.

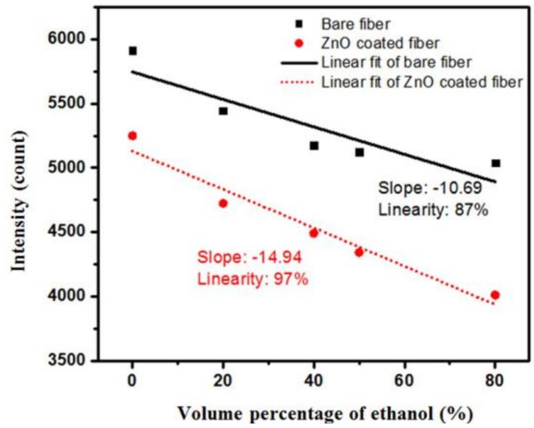


Fig. 6. Output intensity variation against ethanol concentration.

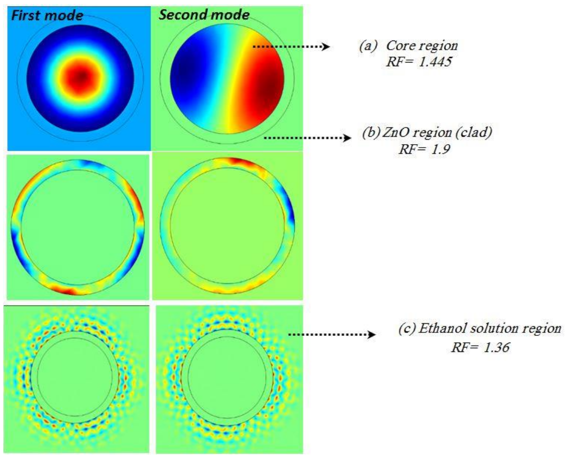


Fig. 8. Numerical simulation of light modes distribution in: (a) core, (b) clad, (c) ethanol solution regions.

In order to investigate light scattering through ZnO nanorods, which is practically illustrated in Fig. 1, we have simulated light modes distribution in the core, clad and solution regions. The optical properties of fiber were studied through a two-dimensional finite element method. The information about fiber structure used for simulation is as follows. The diameter of fiber core is 28 µm. The refractive index of the fiber core and ZnO layer (thin ring with 2µm thickness around the core, defining clad) were considered 1.445 and 1.9, respectively. The refractive index related to different concentration of ethanol was measured experimentally through Abbe optical refractometer and was plotted in Fig. 7.

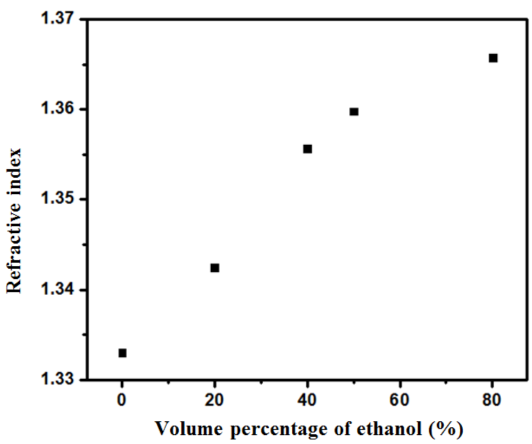


Fig. **7**. Refractive index of different concentration of ethanol measured through Abbe refractometer.

The measured refractive index of the solution was varied from about 1.33 to 1.37 against 0 to 80 volume percentage of ethanol in distilled water. Accordingly, these measured refractive indices can be treated as the initial refractive index values for defining the solution media in the simulation. The two fundamental modes in different regions were shown in Fig. 8. It is clearly seen that the light leaks out near fiber surface and penetrating to ambient medium giving rise to the reduction of transmitted output intensity.

IV. Conclusion

In summary, the effect of etching fiber and growth of nanorods in the magnitude of generated evanescent field was demonstrated experimentally. Well-ordered and relatively high density of ZnO nanorods were grown successfully on the fiber with aspect ratio more than 8. Employing equimolar solution of zinc nitrate and hexamine in hydrothermal treatment resulted ZnO nanorods with hexagonal wurtzite structure. PL analysis indicated presence of oxygen vacancy on the surface of grown nanorods that are dominant point defects and play an important role in sensing process, inducing conductivity variations and resulting refractive index changes. Sensing behavior of the bare fiber compared to ZnO coated fiber in response to ethanol solution (10-80% volume percentage). Results revealed that ZnO coated fiber enhanced sensor performances, time response was measured near 0.6 s, sensitivity more than 14.9 (count/%) and high linearity of 97% exhibited proposed structure is attractive for chemical and biological applications. To investigate more precisely the light leakage and light modes distribution under different concentration of ethanol numerical simulation was carried out.

Acknowledgment

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